# New car odor: sensory and molecular characterization of odors and potential sources of smell in the vehicle interior

# Neuwagengeruch: molekular-sensorische Charakterisierung von Gerüchen und potenziellen Geruchsquellen im Fahrzeuginnenraum

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Buchecker F. performed the experimental work, evaluated, and interpreted the data. Buchecker F., Loos H.M. and Buettner A. contributed to the manuscript, conceived, and planned the study.

## Abbreviations

| | |
|---|---|
| 2D-GC-MS | Two-dimensional gas chromatography-mass spectrometry |
| 2D-GC-MS/O | Two-dimensional gas chromatography-mass spectrometry/ olfactometry |
| AEDA | Aroma extract dilution analysis |
| DB-5 | Durabond-5 ((5 %-Phenyl-)methylpolysiloxane) |
| DB-FFAP | Durabond-Free fatty acids and phenols (polyethylene glycol modified with nitroterephthalic acid) |
| FID | Flame ionization detector |
| GC | Gas chromatography |
| GC-MS | Gas chromatography-mass spectrometry |
| GC-O | Gas chromatography-olfactometry |
| h | Hours |
| MS | Mass spectrometry |
| *m/z* | Mass to charge ratio |
| NIST | National Institute of Standards and Technology |
| NMR | Nuclear Magnetic Resonance |
| O | Olfactometry |
| OD | Odor dilution |
| OEDA | Odor extraction dilution analysis |
| OEM | Original equipment manufacturer |
| SAFE | Solvent assisted flavor evaporation |
| SBSE | Stir bar sorptive extraction |
| SHED | Sealed houses for evaporative emissions determination |
| SPME | Solid phase micro extraction |

| | |
|---|---|
| SVOCs | Semi-volatile organic compounds |
| TVOC | Total volatile organic compound |
| v/v | Volume + volume |
| VDA | Verband der Automobilindustrie e.V. |
| VIAQ | Vehicle interior air quality |
| VOCs | Volatile organic compounds |
| VVOCs | Very volatile organic compounds |

## Abstract

The typical new car smell is very distinctive. Depending on vehicle brand and class, this characteristic odor differs in intensity and elicits either approval or rejection among vehicle purchasers. Up to now, only limited information has been available on the molecular composition of the new car odor. Vehicle manufacturers (OEMs) are mainly concerned with the analysis of toxic compounds and (odorless) main components of the gas phase of the vehicle interior. In addition, OEMs usually do not have an appropriate equipment to characterize odorants by instrumental analysis. Therefore, this work deals with the investigation of odors and potential sources of smell in the vehicle interior.

In the first two chapters of this thesis, odor-active compounds of an aqueous cavity preservation were analyzed by means of solvent extraction, gas chromatography-olfactometry (GC-O) and odor extract dilution analysis (OEDA). Odorants of the investigated corrosion inhibitor could enter the vehicle interior via air bridges between the vehicle body and the passenger cabin and could have a considerable influence on the odor. Identification of the potent odors by two-dimensional gas chromatography-mass spectrometry coupled with olfactometry (2D-GC-MS/O) revealed a series of carboxylic acids and lactones as the main odor contributors of the cavity preservation. The hardening of the cavity preservation affected a change of the odor profile due to the evaporation of odor-potent lactones which was confirmed by a descriptive odor profile analysis. A comparison with odorants of individual materials of the vehicle interior investigated in literature revealed that both identified substance classes are present in a wide variety of materials and, consequently, contribute to the odor in the vehicle interior.

In the third chapter of this thesis, odorants of the aqueous cavity preservation were investigated by means of emission test chamber experiments with the aim to obtain further insights into the odorant composition. Since no appropriate method existed to investigate odorants in a gas phase in the form of a liquid odor extract, different sampling strategies, such as chamber air absorption and adsorption, were compared. Subsequent investigations by GC-O and OEDA revealed potent carboxylic acids and lactones in the gas phase of the heated cavity preservation using each sampling method. In addition, more detailed results were obtained in a descriptive odor profile analysis by sample presentation of chamber air in odor bags compared to a presentation in glass vessels according to VDA 270. As a result, the developed methods may be extended for the investigation of odorants in air of big vehicle assemblies or whole vehicle interiors.

In the fourth and the fifth chapter of this thesis, odorants of two new car interiors with different seat covers were analyzed as general proof of principle. For this purpose, air of the vehicle interiors was investigated in a whole vehicle test stand for interior emissions by means of adsorption and subsequent elution of the volatiles. Following GC-O and 2D-GC-MS/O analyses, 39 odorants were identified belonging to various substance classes such as esters, saturated and unsaturated aldehydes, unsaturated ketones, rose ketones, phenol and benzene derivatives, and pyrazines. Furthermore, ten important odorants were quantified which could be detected in a descriptive odor profile analysis. After the initial investigation, both vehicles were used by a customer in his everyday life. At specific time intervals during use, the odorant composition of the vehicle cabins was analyzed by means of sensory evaluation and instrumental analysis after sampling on the test stand for interior emissions. The knowledge of the decay behavior of the odorants allowed further pinpointing of the most important odorants that caused the typical new car odor in the two investigated cars during vehicle usage. Based on these findings, a targeted investigation of individual components regarding the identified odorants and, thus, a reduction of the exposure to smelly substances in the vehicle interior will be significantly facilitated.

## Zusammenfassung

Innenräume von neu produzierten Fahrzeugen weisen den typischen Neuwagengeruch auf. Je nach Automarke und Fahrzeugklasse ist dieser charakteristische Geruch unterschiedlich stark ausgeprägt und findet bei Fahrzeugkäufern sowohl Zustimmung als auch Ablehnung. Bisher ist über die molekulare Zusammensetzung des Neuwagengeruchs nicht viel bekannt, da sich Fahrzeughersteller hauptsächlich mit der Analyse von toxischen Verbindungen und (geruchslosen) Hauptkomponenten der Gasphase beschäftigen. Zudem besitzen Fahrzeughersteller meist keine geeigneten Möglichkeiten, Geruchsstoffe instrumentell-analytisch zu charakterisieren. Diese Arbeit befasste sich deshalb mit der Aufklärung der Geruchsstoffe und möglichen Geruchsquellen im Fahrzeuginnenraum.

In den ersten beiden Teilen dieser Arbeit wurden geruchsaktive Verbindungen einer wässrigen Hohlraumkonservierung mittels Lösungsmittelextraktion und Gaschromatographie-Olfaktometrie (GC-O) untersucht. Die Geruchsstoffe des Korrosionsschutzmittels können über nicht geschlossene Luftbrücken zwischen der Fahrzeugkarosserie und der Passagierkabine in den Fahrzeuginnenraum gelangen und können den Geruch maßgeblich beeinflussen. Identifizierung der Geruchsstoffe mittels zweidimensionaler Gaschromatographie-Massenspektrometrie gekoppelt mit Olfaktometrie (2D-GC-MS/O) zeigte, dass hauptsächlich geruchspotente Carbonsäuren und Lactone für den Geruch verantwortlich waren. Beim Aushärten der Hohlraumkonservierung änderte sich zudem der Geruch durch das Verdampfen von geruchspotenten Lactonen, was mittels einer deskriptiven Geruchsprofilanalyse bestätigt werden konnte. Ein Literaturvergleich mit Geruchsstoffen einzelner Materialien des Fahrzeuginnenraums zeigte, dass beide identifizierten Verbindungsklassen in verschiedensten Materialien vorkommen und deshalb zum Geruch im Fahrzeuginnenraum beitragen.

Im dritten Teil dieser Arbeit wurden die Geruchsstoffe der wässrigen Hohlraumkonservierung mittels einer Emissionsprüfkammer untersucht, um weitere Einblicke in die Geruchsstoffzusammensetzung zu erhalten. Da keine geeignete Methode existierte, geruchsaktive Verbindungen einer Gasphase in Form eines flüssigen Geruchsextraktes zu untersuchen, wurden verschiedene Strategien der Probenahme, wie die Absorption und Adsorption der Kammerluft, miteinander verglichen. Durch eine anschließende GC-O und Geruchsextrakt-Verdünnungsanalyse konnten mittels jeder Methode geruchspotente Carbonsäuren und Lactone in der Gasphase der erwärmten Hohlraumkonservierung identifiziert werden. Bei der Probendarbietung der Kammerluft in Geruchsbeuteln konnten in einer deskriptiven Geruchsprofilanalyse zudem detailliertere Ergebnisse erhalten werden als bei der Probendarbietung in einem Sensorik-Glas gemäß VDA 270. Die entwickelten

Methoden lassen sich daher auf die Untersuchung der Geruchsstoffe in der Luft von großen Fahrzeugbaugruppen oder Fahrzeuginnenräumen erweitern.

Im vierten und fünften Teil dieser Arbeit wurden die Geruchsstoffe von zwei Neuwagen mit unterschiedlichen Sitzbezügen analysiert. Dazu wurden mittels Adsorption und anschließender Elution flüchtige Verbindungen der Fahrzeuginnenräume in einem Fahrzeuginnenraumemissionsprüfstand untersucht. Durch GC-O und 2D-GC-MS/O Analysen konnten 39 Geruchsstoffe identifiziert werden, die verschiedensten Substanzklassen wie Estern, gesättigten und ungesättigten Aldehyden, ungesättigten Ketonen, Rosenketonen, Phenol- und Benzolderivaten und Pyrazinen angehörten. Zehn wichtige Geruchsstoffe wurden zudem quantifiziert und konnten sensorisch in einer deskriptiven Geruchsprofilanalyse erfasst werden. Beide Fahrzeuge wurden nach der initialen Untersuchung von einem Kunden im Alltag benutzt. In bestimmten Zeitabständen während der Fahrzeugnutzung wurde die Zusammensetzung der Geruchsstoffe in den Fahrzeugkabinen durch sensorische Bewertung und instrumentelle Analyse nach Probenahme am Prüfstand für Fahrzeuginnenraumemissionen analysiert. Durch die Kenntnis des Abklingverhaltens der Geruchsstoffe konnten die wichtigsten geruchspotenten Verbindungen weiter eingegrenzt werden, die während der Fahrzeugnutzung den typischen Neuwagengeruch der untersuchten Fahrzeuge verursacht haben. Eine gezielte Untersuchung einzelner Bauteile bezüglich der identifizierten Geruchsstoffe sowie eine nachhaltige Reduzierung der Belastung des Fahrzeuginnenraums mit Geruchsstoffen wird auf Basis dieser Erkenntnisse wesentlich erleichtert.

# 1 Introduction

## 1.1 Vehicle interior air quality

In everyday life, we are almost constantly in contact with emissions and odorants. Nowadays, people spend most of their time indoors including their own house or apartment, work facility, but also means of transport such as a car [1]. However, the awareness about the quality of the surrounding air has significantly increased in the recent years, as the consciousness of health has changed. Especially unpleasant and untypical odors can lead to concerns about possible toxic effects caused by the product, furnishing or environment [2, 3]. In general, air pollution is recognized as a cause of concern and is associated with serious illnesses, including increased risk of cardiovascular disease, various cancers, and respiratory infections [4]. Additionally, some emitted substances have the potential to cause symptoms such as nausea, allergies, fatigue, burning eyes, and headaches – depending on an individual reaction [5, 6].

In the context of vehicles, emissions are primarily associated with fuels and the related exhaust gases. However, the proportion of components that are not in contact with fuels and may cause emissions is not negligible. In addition to tires, engine components, and materials that are in contact with the outside air, there are also many components inside the vehicle. Over time, emissions from components outside the vehicle are released into the environment; emissions from components inside the vehicle, on the other hand, can accumulate in the passenger cabin [7]. The vehicle interior can therefore be considered as a specific indoor environment [5].

Thus, the specific environment in the passenger cabin and vehicle interior air quality (VIAQ) became an increasing focus for vehicle manufacturers and their customers in recent years. Low emission values are among the most important material properties for automotive interiors, as they contribute significantly to driving comfort. Thereby, the interest surrounding the emissions in the vehicle interior range from regulating compounds that negatively affect health to reducing the typical new car odor, which is too offensive for some consumers. For this reason, components may not release any substances over a long period of time that could be noticeable, for example, due to unpleasant odors, or that could affect the health of the passengers [8, 9].

In addition to the reduction of fuel-based emissions such as $CO_2$ and $NO_X$, the automotive industry has consequently been spending an immense effort on the reduction of outgassing volatile organic compounds (VOCs) in the vehicle interior and improving VIAQ in recent years. Manufacturers and governments have created standards and regulations to monitor a range of (toxic) VOCs in the interior of new cars [10]. The exposure of car users with emissions caused by components in the passenger cabin has consequently decreased in recent years [10].

However, VIAQ is not only influenced by VOCs from various materials in the vehicle interior (see Figure 1). The passengers can also have a significant impact on the quality of the interior air and odor during vehicle usage, and the ambient air of the environment where the vehicle is moved is also an important factor for the quality in the vehicles [9, 11]. At the time of vehicle delivery, VOCs from materials have the most significant influence on VIAQ [12]. However, depending on the usage behavior of the drivers and passengers, these emissions decrease significantly within the first weeks of usage and the passengers determine the quality of the air in the vehicle interior. After only a few months of vehicle usage, individual influences of the passengers and ambient air presented in Figure 1 could dominate the VIAQ, depending on their users and the environment where the vehicle is moved [12].

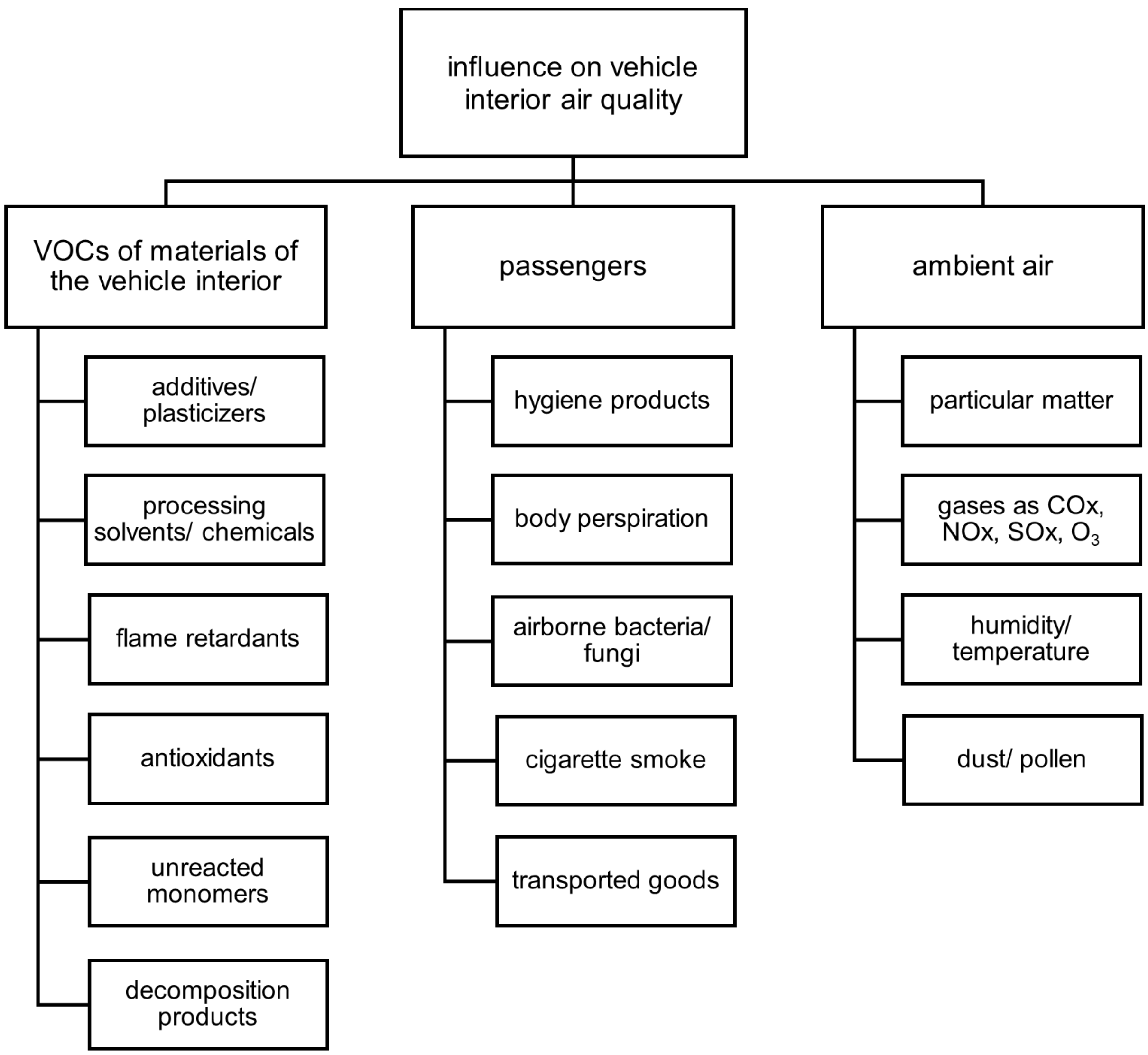


**Figure 1:** Influences on vehicle interior air quality. Data for image: [1, 8, 10, 11, 13–15].

Besides the modification of the components and materials in the vehicle interior regarding their emissions, vehicle manufacturers are also working on intelligent and automated ventilation concepts or are improving the vehicle structural and sealing design which can positively impact the VIAQ [16]. Permanent recirculation of the air conditioning system can reduce the accumulation of $CO_2$ in the vehicle, and targeted control of the intrusion of external air or the ventilation rate can reduce particle concentrations [16, 17]. By closing air bridges between the vehicle body and the interior, penetration of volatiles into the passenger compartment can be additionally prevented [16]. However, it is difficult for vehicle manufacturers to influence the strength of the influence of passengers and ambient air on the VIAQ.

Additionally, the geographical origin of vehicle customers also has a great impact on the perception of the VIAQ. The awareness of bad air and air pollution is more pronounced in Asia than in the rest of the world. This awareness is not only limited to the vehicle interior, but also extends to products and enclosed rooms [18]. Especially in buildings, emissions can accumulate which can affect people stronger due to lower air exchange rates and the lack of filters in fresh air supply compared to vehicle interiors. However, people who are very sensitive to indoor air pollution may therefore develop a so-called sick-building syndrome [19]. Since people nowadays also spend a lot of time in vehicles, it is possible that the passenger compartment can also cause this disease.

Furthermore, odorants have a great impact on the perception and evaluation of the quality of the perceived air and have a direct influence on customers' buying decisions [20]. If a vehicle cabin provides an unpleasant odor or offers poor olfactory VIAQ, customers directly associate their well-being with a lower level of comfort. However, since comfort plays an important role in the customer experience during vehicle purchase, knowledge of the odorant composition is of great importance [21]. Additionally, a too strong odor or especially a “bad” odor is associated by the customer with a health hazard and should therefore be minimized. To address the customers’ expectations of a nearly odorless vehicle interior, it is first necessary to identify the emission sources. Consequently, the focus of the automotive industry is on the design of lower- or zero-emission materials, the continuous monitoring of emissions which have the potential to negatively affect the health of drivers and passengers, and continuous reduction of disturbing substances such as (unpleasant) odorants.

## 1.2 Emissions in the vehicle interior

### 1.2.1 Classification of emissions in the passenger cabin

Organic emissions released from materials in the vehicle interior can be divided into three different groups based on their boiling points (see Table 1) [10]. Very volatile organic compounds (VVOCs) such as solvent residues, additives, and monomers can be gaseous at room temperature and their release into the passenger cabin can be observed immediately after vehicle assembly. Semi-volatile organic compounds (SVOCs) such as long-chain alkanes, fatty acids, and phthalates, on the other hand, are mainly released from materials during elevated temperatures. SVOCs are steadily emitted into the vehicle interior for several years and the concentration is usually several orders lower than VVOCs [5]. SVOCs are also known as compounds causing the "fogging" phenomenon. Thereby, semi-volatile compounds re-adsorb on cold surfaces such as the windshield after the passenger cabin has cooled down, causing a gray (fog) film [22].

**Table 1:** Classification of VOCs in the vehicle interior based on their boiling points. Data for table: [5].

| Group of compounds | Abbreviation | Boiling point |
|---|---|---|
| Very volatile organic compounds | VVOCs | < 0 °C to 50 - 100 °C |
| Volatile organic compounds | VOCs | 50 - 100 °C to 240 - 260 °C |
| Semi-volatile organic compounds | SVOCs | 240 - 260 °C to 380 - 400 °C |

The quantitatively dominating group of emissions in vehicle interior are VOCs. Numerous studies showed that these compounds are mainly composed of non-polar aromatic, aliphatic, and saturated or unsaturated (cyclic) hydrocarbons [7, 13, 14, 23–25]. Thereby, several hundred different substances can be present in the passenger compartment [5]. Semi-polar compounds such as alcohols and carbonyls are usually found only in small amounts with less than 10 % in vehicle interiors [26]. Additionally, odorants of the vehicle interior mainly belong to the group of VOCs, however, they are among the semi-polar compounds occurring quantitatively in very low concentrations [27]. Depending on the material properties, VOCs are also released into the interior, sometimes over several years [12]. Especially materials, that are not exposed to direct sunlight or intense heat, release emissions mainly in a diffusion-controlled manner into the interior or are products of degradation, hydrolysis or oxidation reactions [12].

### 1.2.2 Emission sources of the car interior

Emission sources in the vehicle interior are very diverse. Predominantly, materials equipped in the passenger compartment release VOCs, whereby a variety of different materials and components can emit the same volatiles, complicating the identification of the emission sources [5]. The origin of organic emissions can also be metallic or inorganic materials, which may gradually release volatile compounds during vehicle usage due to VOC accumulation prior to the assembly of the material [5]. In addition, porous materials installed in passenger cabins can serve as sorbents, allowing the adsorption of any released VOCs and subsequent stepwise desorption [28, 29].

Plastics are the most important class of materials contributing to emissions in the vehicle interior. Polyethylene, polypropylene, polystyrene, polyamide, polyester, acrylonitrile-butadiene-styrene, polyurethane, and others are widely used in the passenger compartment due to their almost unlimited possibility of formability, low weight and good availability [5]. Due to their applications in air conditioning systems, ceiling trims, trunk shelves, door and instrument panels, carpets, and seats, plastics constitute the largest surface area in vehicle interiors and thus provide a significant source of VOCs [5]. Consequently, a large number of studies of their volatile compounds can be found in the literature [30–33]. However, the volatiles of plastics are mainly degradation products, additives, processing solvents and chemicals, flame retardants, antioxidants and to a large extent unreacted monomers [34]. In recent years, the odorants of plastics have increasingly become the focus of analysis [35]. The continuous development and use of recycled plastics requires investigation and improvement of these materials, especially with regard to their contamination with odor-active compounds [36, 37].

Another material with a large surface in vehicle interiors provides leather. While efforts have been made to reduce interior emissions of VOCs or odor-active substances, the smell of leather in vehicle interiors is often desirable, at least in Europe and America [38]. In Asia, on the other hand, and even increasingly in the rest of the world, leather is being replaced with artificial leather made of polyvinyl chloride or polyurethane, as these have lower emissions and are more accepted by customers [39]. In addition to the visible materials of the vehicle interior, other auxiliaries such as lubricants and adhesives are applied during vehicle assembly. These types of materials can also contribute to VOC emissions in the passenger cabin [40]. Although the quantities of these constituents are not high and the surface areas are small compared to plastics, odor-active emissions from these materials can have a significant impact on VIAQ [27]. In addition, there are other components in the passenger cabin that can release emissions, such as rubber, natural fibers, and wood [41, 42].

### 1.2.3 Legal framework and regulatory requirements

Besides emissions that are harmless to human health and the environment, toxic VOCs can also be found in vehicle interiors [17]. For this reason, OEMs conduct VOC screenings of the interior air of vehicles and of the gas phase of components, assemblies, and individual materials [43]. During these experiments, air is adsorbed on a suitable sorbent and gas chromatography-mass spectrometry (GC-MS) analyses are applied to identify and quantify the emissions [44]. On the other hand, the overall amount of all volatile organic compounds is quantified and reported as the TVOC concentration (total volatile organic compound), allowing a brief assessment of the general air quality [5]. In this context, however, there are only recommendations and no scientific confirmations available. Therefore, it is difficult to establish precise guidelines or legal requirements since vehicles are exposed to higher air exchange rates during driving compared to closed rooms in buildings [45]. For specific toxic compounds, however, there are guidelines or regulations in three Asian vehicle markets (see Table 2) [46–48]. In Korea and China, mandatory regulations have been established which OEMs have to fulfill. In Japan, on the other hand, a voluntary guideline for reducing vehicle cabin VOC concentration levels has been developed.

**Table 2:** Overview of limitations of individual analytes [µg/m³] in the vehicle interior air in Japan, Korea, and China. Data for table: [46–48].

| **Analytes** | **Japan** | **Korea** | **China** |
|---|---|---|---|
| Formaldehyde | 100 | 210 | 100 |
| Acetaldehyde | 48 | - | 50 |
| Acrolein | - | 50 | 50 |
| Benzene | - | 30 | 110 |
| Toluene | 260 | 1000 | 1100 |
| Xylene | 870 | 870 | 1500 |
| Ethyl benzene | 3800 | 1000 | 1500 |
| Styrene | 220 | 220 | 260 |
| Di-*n*-butyl phthalate | 220 | - | - |
| Di-2-ethylhexyl phthalate | 120 | - | - |
| *n*-Tetradecane | 330 | - | - |
| *p*-Dichlorobenzene | 240 | - | - |

In the three Asian markets, the compounds formaldehyde, acetaldehyde, ethylbenzene, styrene, toluene, and xylenes are regulated with respect to their concentrations in the vehicle interior. The problem is, however, that the regulations or guideline specify sampling at various temperatures and differences appear in the test method details [5]. Consequently, one standardized test for the emissions in the vehicle cabin is not sufficient. For this reason, the DIN ISO 12219 standard was developed in recent years to harmonize the test procedures describing normalized tests on interior air quality based on the simulation of three vehicle modes (see chapter 1.3) [49]. The aim was to avoid that OEMs control the VIAQ based on their own standards and to ensure that the results are comparable. Additionally, the implementation of an worldwide accepted testing procedure would allow an independent quality control and testing, and the process of vehicle or material approval on markets with different legal regulations may be significantly simplified [5].

### 1.2.4 Odor-active compounds and exposure in automotive mobility

Only a small amount of the vehicle interior emissions is odor-active [13, 14]. However, the substance group of odorants in vehicles has a great influence on the purchasing behavior of customers and on the acceptance of vehicles. One reason is that the odor is considered to be the sense that is most closely related to emotions as the olfactory system for perceiving volatile substances is directly linked to the limbic system [50, 51]. The perception of an odor is accompanied by an emotional evaluation and is also considered to be a warning system [52]. Consequently, if a vehicle smells too strong or is not pleasant for the customer, this might suggest a health hazard and may prevent a purchase, especially in the case in which customers perceive irritating sensation. Likewise, if a passenger cabin smells too weak or neutral, or unlike the customers' expectations especially in relation to being newly produced, the odor can influence the purchase decision [20, 52, 53]. Accordingly, several OEMs integrate new car odors as part of their marketing strategy, thus creating an association among potential customers and the vehicle make with a particular new car odor [54].

A completely odorless new car is not necessarily well received by customers, as can be seen in the marketing strategies of various OEMs. Manufacturers of luxury vehicles such as Rolls-Royce Motor Cars responded to customer complaints that their new cars offered a too strong plastic-like odor [54]. As a result, leather-like smelling odorants were actively introduced into the seat trim, which was considered to be of high value by the customers [54]. The fact that the new car smell has a significant role in the marketing is also demonstrated by the American car manufacturer Cadillac, which developed a special new car smell called "Nuance" especially for its vehicles. Thereby, the manufacturer achieved that the customers associated this brand with a distinctive smell [54]. Another way of influencing the odor of the vehicle interior is offered by optional extras during vehicle purchase. Instead of the conventional use of a fragrance infuser by the customer, active scenting by equipment integrated into the vehicle's ventilation system is offered by OEMs, especially in the segment of luxury cars [55]. This allows customers to choose between various fragrances and to influence the odor of the vehicle cabin according to their own preferences.

The use of odors in vehicle interiors, however, can also cause negative effects. Some people perceive these odors as "too much" and the exposure to these scents can thus become a burden. Some odor-active substances can cause headaches, irritation of the mucous membranes, respiratory problems, or allergies. As a consequence, some people fundamentally reject any non-natural odorants [56]. This phenomenon is particularly pronounced in the Asian market. Therefore, OEMs perform considerable efforts to provide a nearly odorless vehicle to these customers and to obtain deeper insights into the odorant composition within the vehicle interiors.

So far, the specific molecular composition of the new car odor was not fully resolved. Previous studies focused primarily on the analysis of VOCs by GC-MS, and odorants were only identified if they were quantitatively detectable via standard analyses [9]. Therefore, the knowledge about odor-causing trace components in the vehicle interior is not existent. A possibility to detect odorants analytically is offered by advanced sensory analysis methods that combine GC analysis with human sensory evaluation, such as gas chromatography-olfactometry (GC-O, see chapter 1.5.2) [57]. This method has primarily been used for the analysis of aroma compounds in foods and beverages and has recently been extended to non-food products such as plastics and wood [58, 59]. However, this method has also been successfully used for the analysis of odorants of a small number of materials of the vehicle interior such as plastics and seat covers [27, 34, 39, 60]. An overview of these materials and their most important odorants is given in chapter 3. In this context, most of the odor-active compounds could be assigned to the substance class of (poly)unsaturated carbonyl compounds or derivatives of phenol or guaiacol [61]. Thereby, the odorants originated from contaminated raw materials or were formed during production or storage via thermal degradation or oxidation processes [62].

## 1.3 Investigation of vehicle interior air emissions

### 1.3.1 Whole vehicle test stand for vehicle interior emissions

As emissions originating from passengers and the environment of the vehicle interior can have a significant impact on the air quality in the passenger cabin, it is necessary to comply with special requirements for measurements of the vehicle interior emissions. Especially for investigations of toxic substances, the evidence for type approvals of vehicles, and compliance with legal requirements, it is important to ensure that compounds can unambiguously be attributed to the passenger compartment [49]. Furthermore, it is essential that analyses of vehicle interiors are comparable, repeatable, transparent, and verifiable [49]. For this reason, measurements of volatile compounds of the vehicle interior by OEMs are carried out in specially designed whole vehicle test chambers or whole vehicle test stands (see Figure 2).

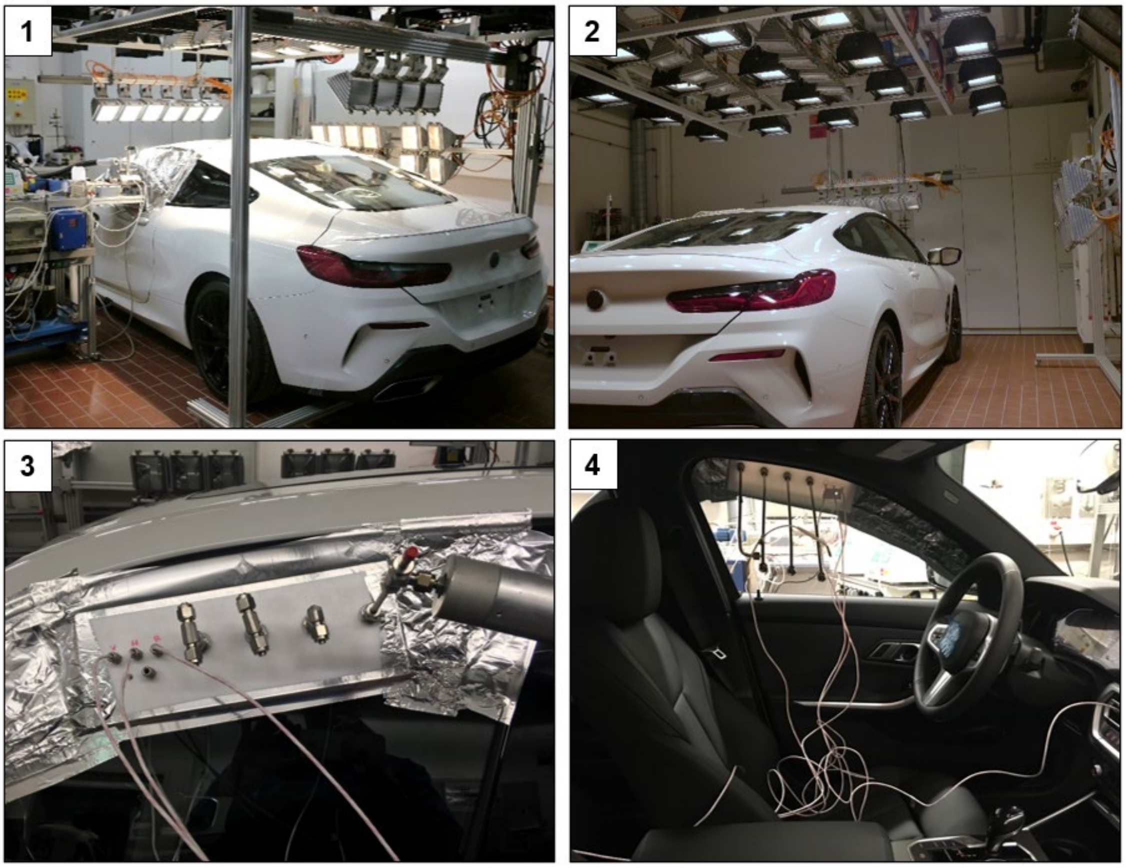

**Figure 2:** Heating a vehicle in a whole vehicle test stand for interior emissions (no. 1 and 2) with an installed sampling interface replacing the window on the front left side (no. 3) and temperature sensors and sampling tubes at the head level of the driver in the passenger cabin (no. 4). Printed with permission of BMW Group.

Requirements of a whole vehicle test stand include the ability to adjust the temperature and humidity of the entire test stand and to reduce the background concentration of volatile compounds to a minimum [49]. For this purpose, a supply of purified neutral air is provided to reduce unwanted background emissions in the test chamber [49]. In addition, a controlled heating of the passenger compartment must be possible, since regulatory guidelines require the knowledge of emissions at elevated temperatures. Furthermore, the contamination of the passenger cabin with VOCs is greatly increased at higher temperatures [63]. For this reason, temperature sensors are installed at various positions in the vehicle, such as the instrument panel, parcel shelf, driver's head, etc. (see Figure 2 no. 4) [49]. However, depending on the test methods, two different heating procedures are applicable.

During sampling at a specific temperature, the passenger compartment is heated using radiant heaters above, in front and at the side of the vehicle (see Figure 2 no. 1). In this case, worst-case scenarios can be simulated, in so far as the vehicle interior may be heated up to 65 °C. However, these procedures extend over different time periods depending on the vehicle model, size, and equipment, and may thus cause variable effects [64]. Individual materials of the vehicle interior that are exposed to the direct heat of the radiant heaters can subsequently reach temperatures of more than 100 °C. Thereby, the temperature at the driver's head position serves as a reference temperature [64].

Furthermore, sampling of the vehicle interior emissions after heating with a constant radiation power is possible. In this case, the intention is to simulate parking of the vehicle in the sun, which allows realistic and exposure-related measurements of the vehicle interior emissions [49]. Height-adjustable radiant heaters can be set above the vehicle to achieve a constant radiant power on the roof surface of vehicles with different heights (see Figure 2 no. 2) [49]. Vehicles with a small interior heat up faster during the same test period than, for example, an SUV with a large interior volume [10]. However, the test method according to DIN ISO 12219-1 considers heating with a constant radiation density on the roof surface of 400 W/m² [49]. To sample and detect the VOCs as realistically as possible, this method regulates sampling of air from the vehicle interior at different times during the measurement. However, sampling is possible by replacing a window with a sampling interface with fittings for sensors and pumps (see Figure 2 no. 3) [49].

The sampling procedure according to DIN ISO 12219-1 is classified according to three different modes (see Figure 3). After conditioning the test stand or test chamber, the vehicle interior is preconditioned. Thereby, the doors of the vehicle are opened for 1 h allowing the passenger compartment to acclimate to the temperature of the test stand at 23 °C. Afterwards, the doors are closed for at least 8 h so that volatile compounds can accumulate in the gas phase of the passenger cabin. At the end of this so-called ambient mode, sampling takes place.

Subsequently, the parking mode is simulated by heating the vehicle interior for 4 h, which is directly followed by the driving mode. Here a door is opened, the engine is started, and the air conditioning system is turned on in less than 60 seconds; at the same time the pumps for sampling are started. The objective here is to simulate the customer's behavior when entering a heated vehicle. During all sampling steps, additional blank samples of air in the test stand are analyzed [49].

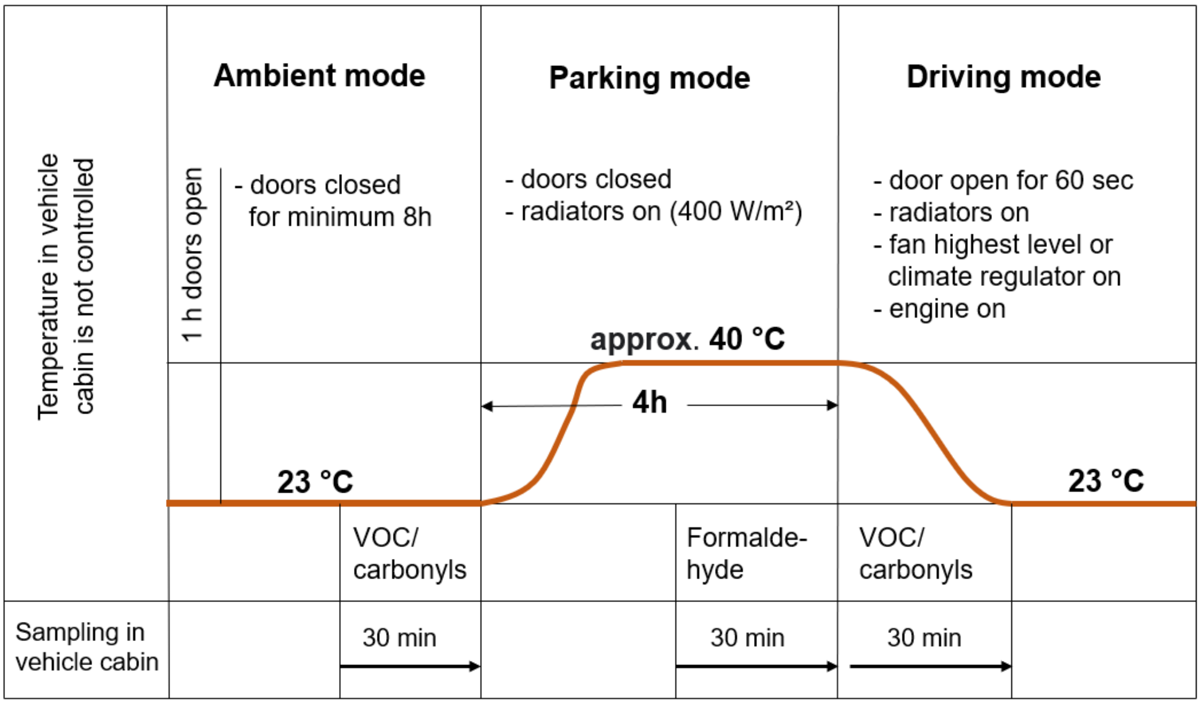


**Figure 3:** Sampling procedure of an interior air emission measurement in a whole vehicle test stand according to ISO 12219-1. Redrawn and adapted from [49].

### 1.3.2 Emission test chambers

Individual materials or assemblies of vehicle interiors can be examined to determine their released emissions analogous to the examination of whole vehicle interiors. Emission test chambers, also known as SHED chambers (sealed houses for evaporative emissions determination), are used for this purpose (see Figure 4 no. 1) [65]. Thereby, an object is placed in an approximately ideally mixed test chamber and stored at a specified temperature, humidity, and air exchange rate (see Figure 4 no. 2) [33, 66]. Due to the different volumes of test objects, different chamber sizes with different test space volumes may be used. Small test objects are investigated in a test chamber with a volume of 0.24 m³, larger objects are analyzed in chambers with a volume of 0.98 m³ to 4 m³. VOCs released from the materials or assemblies accumulate in the test chamber and are discharged via an air stream. To minimize interface effects as adsorption or condensation processes, the chamber and all components in contact with the chamber air are made of stainless steel. Air samples can be collected at various times using a heated sampling tree (see Figure 4 no. 3 and 4) [67].

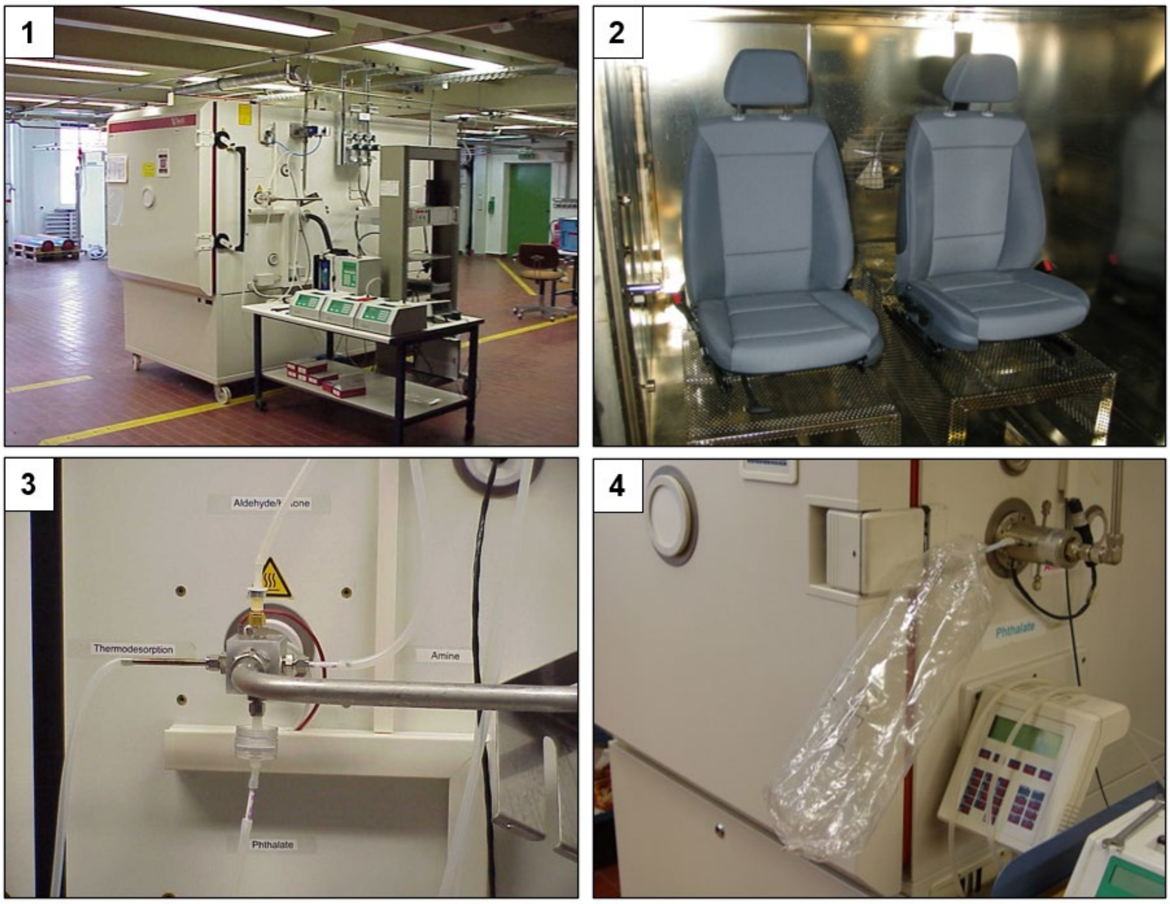


**Figure 4:** Emission test chamber with peripheral equipment (online FID, gas pipelines, and pumps; no. 1) with seats inside the test chamber (no. 2) and a detailed view of the heated sampling tree with connected specimens (no 3 and 4). Printed with permission of BMW Group.

Investigations of materials and assemblies of the vehicle interior are carried out according to DIN ISO 12219-3 to -5, and modified protocols thereof. Thereby, the materials are heated to 65 °C and air samples are collected within 4.5 h [67]. As a result, qualitative and quantitative data of VOCs and special compound classes such as amines and carbonyls can be determined. Likewise, chamber air can be sampled to evaluate the odor [68]. Additionally, the obtained emission data can be used to establish a correlation between concentration levels in the material and in the vehicle interior [67]. However, the measurements are mainly carried out for the qualification of newly designed and developed interior components with changed material compositions [23].

Prior to the investigation, the storage of the materials or components is of decisive importance. Compounds from the environment and ambient air, that were originally not represented in the sample, can adsorb to the test object. In addition, each component must be carefully protected against chemical contamination or physical influences such as heat, sunlight, and moisture until the beginning of the investigation [67]. For this reason, test objects are sealed in aluminum foil or polyethylene bags after disassembly or production and, thus, are protected from ambient air and other contact [67]. Prior to the emission test chamber experiment, the samples must be stored exposed to ambient air for seven days. However, if packaged samples are analyzed immediately after opening, additional compounds which adsorbed on the surface during storage can be directly analyzed [69].

## 1.4 Human sensory evaluation methods for the characterization of automotive products

Human sensory evaluation methods are used since decades for the assessment of the odor in vehicle interiors or components and materials of the passenger cabin. Compared to the instrumental-analytical investigation, human sensory evaluation methods are an easy and efficient possibility to analyze samples [70]. In addition, a rapid estimation is possible whether an odor optimization is necessary or not. In this respect, methods have been established in the automotive industry allowing the rating of intensity and hedonic. The respective vehicle manufacturers either directly adopt standardized norms from VDA, ISO as well as DIN or implement these norms in a (slightly) modified form according to their own test specifications [71, 72].

For human sensory evaluations, however, the exposure of humans to toxic substances must be considered and should be reduced to a minimum. Prior to olfactory analysis, samples must be analyzed regarding to substances that are hazardous to health and, in the case of a known concentration of the components, the applicable occupational exposure limits in relation to intended dilutions must be determined and respected [73]. In case of suspicion that panelists are exposed to such substances, they must be informed about these risks [73].

A method for testing materials and smaller components is described in the German VDA 270. In this test, a certain amount of a material is stored in a glass vessel representing a defined volume for a certain time period and temperature, and in the presence or absence of humidity [71]. Generally, common preserving jars are used as glass vessels in this method. The odor of the headspace is subsequently rated with grades from 1 (no odor perceptible) to 6 (odor is not acceptable). At least three sensory participants evaluate the odor, although they do not necessarily have to be trained or be experienced in sensory evaluation [71].

Another human sensory evaluation method of automotive products is performed by means of odor bags, as described in ISO 12219-7. In this case, air from emission test chambers or vehicle interiors is filled into airtight, odorless, non-permeable, and non-adsorptive air sampling bags [72, 74]. Thereby, air is sampled into an empty bag by applying negative pressure according to the working principle of the lung with the use of an odor sampling device [72]. By means of an odor presentation device such as the PureSniff device from Olfasense (see Figure 5 right), each panelist receives an identical sample with a standardized volume flow and a constant provision time. For this purpose, the odor bag is placed in a cylinder, which can increase the pressure with a controlled injection of clean air. Air from the bag is then transferred to the nose by means of a funnel [75]. The odor is evaluated similar to VDA 270 [72]. In addition, odor pleasantness or olfactory notes can be assessed depending on the experimental procedure and purpose. For this evaluation method, at least five trained panelists are required [72].

**Figure 5:** Olfactometer (left) and odor presentation device for air sampling bags with gas connections (right). Printed with permission of BMW Group.

An additional human sensory approach for analyzing odor samples in bags is described in DIN EN 13725 and is carried out using an olfactometer (see Figure 5 left). By means of dynamic olfactometry, odorant concentrations of pure substances and defined mixtures can be determined in air sampling bags [73]. By stepwise dilution of odor samples with unknown composition with odorless, neutral air, the concentration can additionally be specified as odor unit per cubic meter ($OU/m^3$) [73, 76]. The odor concentration is quantified by determining the dilution factor at which 50 % of the assessors perceive an odor. At least four trained panelists evaluate odor in this method [77]. Panelists are considered as trained by proving a mean odor threshold concentration for *n*-butanol of 62 - 246 $\mu g/m^3$ and a standard deviation of less than 2.3 for at least 10 replicate measurements [73, 78–80].

Human sensory methods according to VDA 270, ISO 12219-7 and DIN EN 13725 represent rapid and simple test procedures, which can be carried out very easily to some extent. However, one general weakness of these methods is the lack of training or comprehensive testing of the olfactory capabilities of the panelists. Consequently, the application of the methods for targeted odor optimization is limited, since primarily intensity and hedonics are evaluated.

Descriptive sensory methods such as odor profile analysis according to ISO 13299 allow a comprehensive approach by describing the predominant odors, as shown in chapters 4 - 7 [70, 81]. During an odor profile analysis, first, odor qualities are determined, and second, intensities of each of the defined odor impressions are evaluated. The individual results are averaged and are usually presented in the form of a spider-web diagram [82]. In addition, the hedonic impression can be evaluated, which is usually done on a scale from 0 (dislike) to 5 (neutral) and 10 (like) [34]. Furthermore, the odor profile analysis enables the perception of a specific odor difference of two or more samples by indicating the presence or absence of specific substance classes or single odorants [82]. In the case of complex odor mixtures, however, individual odor descriptions cannot necessarily be assigned to individual compounds, since several individual odorants can be responsible for the respective odor impression due to additive, suppressive or synergistic effects [83, 84]. Therefore, instrumental analytical methods are used to characterize the underlying odor-active substances systematically. However, the effort of an odor profile analysis cannot be compared to the previously mentioned human sensory methods as a minimum of eight panelists have to be trained and motivated and have to practice the perception of individual odorants on a regular basis [70].

## 1.5 Instrumental analytical methods for the characterization of odorants

### 1.5.1 Isolation, sampling, and concentration methods for volatile compounds

Sampling, isolation, and concentration of volatile compounds for instrumental characterization are of central importance and great challenge in analytical chemistry. Depending on the sample matrix and the expected analytes, different work-up procedures are required. Emissions and especially odorants are characterized by a large chemical diversity, accordingly an appropriate method is required to consider different substance properties like polarity, solubility, and volatility [85]. Some analytes, for example, have very low boiling points, whereas other substances are very difficult to evaporate but are still of analytical interest. Furthermore, very volatile analytes can decompose during sample workup or injection at high temperatures and can form unwanted artefacts [86]. In addition, different concentration levels of target compounds must be considered during sampling, so either an instrumental separation is possible, or an enrichment of target analytes present in very low concentrations can be accomplished. However, sample preparation should also consider matrix effects, which can lead to artifact formation when non-volatile components interfere with the isolation of volatile substances [87].

For the analysis of volatile compounds or odorants in vehicle interiors or related components and materials, basically two different sampling approaches are possible. On the one hand, the gas phase of the vehicle interior air or the headspace above components and materials can be analyzed directly or after enrichment, and on the other hand, volatile compounds and odorants can be extracted with the aid of solvents [44, 66, 88]. Many methods have been established over the years. Albeit, the analysis or extraction method needs to be carefully selected for each analytical task [89, 90]. Selected methods of both general approaches (analysis of gas samples and solvent assisted extraction) will be discussed in more detail in the following.

**Methods for gas sampling**

Methods for gas sampling can be classified into active and passive sampling [91]. In the case of active sampling, a pump is required to suck air through a suitable collection phase, which can be further analyzed with or without enrichment. In this context, the pump is of particular importance since sampling requires the exact determination of the collected air volume for quantitative results [92]. Usually, air volumes are specified in standard liters for the applied pumps, which are valid for conditions at 20 °C and an air pressure of 1013 mbar. However, pumps also allow conversion for sampling under different conditions. In the case of passive sampling, on the other hand, volatile compounds accumulate on/ in a medium by diffusion or flow through a suitable collection phase [93].

**Active enrichment of air** represents one possibility for gas sampling. The most commonly used option in this respect is the **adsorption** of emissions and odorants onto suitable adsorbents such as porous polymers, molecular sieves, or carbon blacks [94, 95]. Thereby, volatile compounds accumulate on materials with a large surface area and adsorb physically by means of nonspecific, nondirectional van der Walls' forces depending on their polarizability, molecular weight, and geometric structure [96]. Depending on the analytical task, an appropriate adsorbent is used allowing an almost complete recovery for the target analytes. The market for adsorbents offers a wide range of materials for this purpose, including materials that adsorb, on the one hand, very volatile compounds with less than three carbon atoms or, on the other hand, heavy molecules with more than 20 carbon atoms by providing the possibility of selective enrichment of individual substance classes [95]. Carriers are usually tubes or cartridges made of glass, plastic, or metal in which small amounts of adsorbents are embedded. During sampling, the flow and volume of air is essential to ensure optimal interaction between analytes and adsorbents and to avoid breakthrough of volatile compounds [97]. However, in the context of vehicle interior emissions, the adsorbents Tenax® TA and Carbotrap® 300 are commonly used as they adsorb volatiles with carbon atoms in the range of C5 and C2 to C20, respectively [23, 98]. The adsorbed VOCs are desorbed mainly by thermodesorption, using a carrier gas and heat to focus emissions on a cooling trap [99]. Another possibility of desorption is offered by solvent desorption, which can be used for targeted analysis [100].

Another possibility of active enrichment of air is offered by **absorption**. Volatile compounds can be dissolved and enriched by means of solvents in washing bottles. However, this method requires a sufficiently high concentration of some analytes or an additional concentration step for the detection of trace compounds, which may lead to evaporation of very volatile compounds or co-elution with the solvent [100]. A more frequently used method is sampling by using **chemical reactions**. In this case, the focus of sampling is on specific substance classes. This sampling approach is applied when a certain group of substances is reactive and a conversion into a more stable form is possible. Target compounds can be separated from the matrix by specific reactions and enabling detection even if their concentration is very low [101]. In the field of routine analysis of emissions in vehicle interiors and from components, targeted derivatization of aldehydes and ketones with dinitro phenyl hydrazine is applied [102].

In odor and VOC research, numerous individual methods have been developed that enable, among other things, the quantification of unstable sulfur compounds [103].

In contrast to the previously mentioned methods, **active sampling without enrichment** allows only limited trace analysis and the investigation of odorants is very difficult, as especially these compounds usually occur in low concentrations [57]. **Sampling in bags** is an option if air has to be analyzed at very inaccessible places, however, bags can be connected directly to a laboratory instrument or sampled on adsorbents [104]. More frequently, instruments or sensors are applied for the **in-situ analysis** of emissions. The spectrum of applications for gas sensors is broad and depends on the analytical task as well as the use of online instruments such as MS or a flame ionization detector (FID) [105–107]. In this context, it should be pointed out that especially online FID are used for emission measurements of vehicle interiors and emission test chambers [49, 67, 100]. They allow direct observation of the TVOC concentration and enable calculation of recovery rates in thermodesorption analyses. In addition, an FID value allows to estimate the volume of air which is necessary for sampling to avoid breakthroughs for adsorption or to obtain a sufficiently high concentration. Furthermore, an FID can be applied for monitoring the room air, which would have a direct impact on the results if high levels of volatile compounds are present [69].

Two other possibilities for **passive sampling** of gas samples are **solid phase micro extraction** (SPME) and **stir bar sorptive extraction** (SBSE). In the case of SPME, a fiber is provided to adsorb volatile compounds, which can subsequently be injected into a gas chromatograph by means of thermodesorption. The fiber material consists of a short, fused silica fiber coated with various materials, typically polydimethylsiloxane and polyacrylate [108, 109]. Sampling via SBSE is based on the same method: a stir bar with a surface of polydimethylsiloxane adsorbs volatile compounds, which can be desorbed by a special desorption unit [110]. Additionally, both adsorbents can also be immersed in solvents [110, 111]. However, in the automotive sector, these methods are not widely used for routine measurements since the concentrations of VOCs in vehicle interiors are occasionally too low [112]. For analyses of individual materials and small samples, on the other hand, applications can be found in the literature [111, 113–115].

Three further possibilities for the investigation of volatile compounds of (small) materials are **direct thermodesorption** and **static and dynamic headspace** measurements. During direct thermodesorption, small materials are placed in empty adsorption tubes and are desorbed and injected in a GC system [116]. In static headspace measurements, a sample is placed in a small container which can be heated under controlled conditions. After equilibrium of the volatiles of the sample with the gas phase in the container, a gas sample can be collected via a septum and injected into a gas chromatograph [117]. In the case of dynamic headspace sampling, the gas phase of a material in a container is collected by means of an inert carrier gas stream and enriched on a cryogenic trap or adsorption material. The trapped analytes can then be desorbed and injected in a gas chromatograph or extracted using a solvent [118]. All methods are suitable for an efficient screening of volatile compounds and are easy to implement, however, the samples have to be representative and homogenously grindable [118].

**Solvent assisted extraction of volatiles**

Extraction of volatile compounds from components and materials of the vehicle interior by means of solvents is mainly used for the preparation of odor extracts. Homogeneously ground and representative samples are added to solvents such as pentane, diethyl ether and dichloromethane, and volatile compounds are extracted (several times) [119]. Since non-volatile matrices can dissolve during this process, further work-up is necessary [109]. State-of-the-art for the isolation of volatile compounds is the solvent assisted flavor evaporation (SAFE). This process involves the evaporation of volatile compounds and solvents under high vacuum at low temperatures [120]. Thereafter, Vigreux distillation and micro-distillation according to Bemelmans are applied to remove the solvent [121]. This method was initially developed for the analysis of odorants in food and beverages. However, there are many studies indicating that this methodology is also applicable for the analysis of volatile compounds and odorants comprised within non-food products and materials of the vehicle interior [40, 41, 58, 59, 61, 122].

Further methods for the analysis of volatiles by means of an extraction process are steam distillation or Soxhlet extraction [108, 123]. However, these methods involve heating the samples at high temperatures, which can cause degradation and formation of artifacts [119]. Therefore, these methods have not been imposed for the analysis of (very) volatile compounds and odorants. Other methods such as simultaneous distillation-extraction or extraction using liquid $CO_2$ are described in the literature but will not be discussed in this thesis [124].

## 1.5.2 Separation and detection methods for odorants

Volatile compounds and odorants are usually separated by means of gas chromatography. As described in chapter 1.5.1, different injection options are available, so that either a gas is injected directly or a liquid is injected, which can be completely vaporized. During gas chromatography, volatiles are separated by passing along a coated capillary column (stationary phase) by means of a constant inert carrier gas flow (mobile phase), whereby the volatile compounds interact with the stationary phase and cause retention of their elution [125]. Detection of VOCs and odorants at the end of the capillary column is usually accomplished by an FID and/ or a mass spectrometer [126]. There are a variety of capillary columns enabling differential separation of the analytes [127]. Additionally, a range of detectors are available allowing the detection of specific substance classes with a higher sensitivity and selectivity [128].

Most of the volatile compounds in vehicle interiors or from components and materials are odorless. However, odorants are usually present only in very low concentrations compared to other odorless emissions [129]. By means of GC-O, odorants can be detected sensitively and selectively among the array of volatiles utilizing the special skills of the human nose [130]. Thereby, the gas effluent is separated at the end of the capillary column: one part is detected with an FID (and/ or MS) and is recorded as a chromatogram, and another part is transferred to a sniffing port (see Figure 6). This allows human assessors to mark odorants in a chromatogram, to describe and record the odor impression and to evaluate the odor intensity [126]. However, the concentration of the perceived odorants during GC-O must be higher than their respective odor threshold [131].

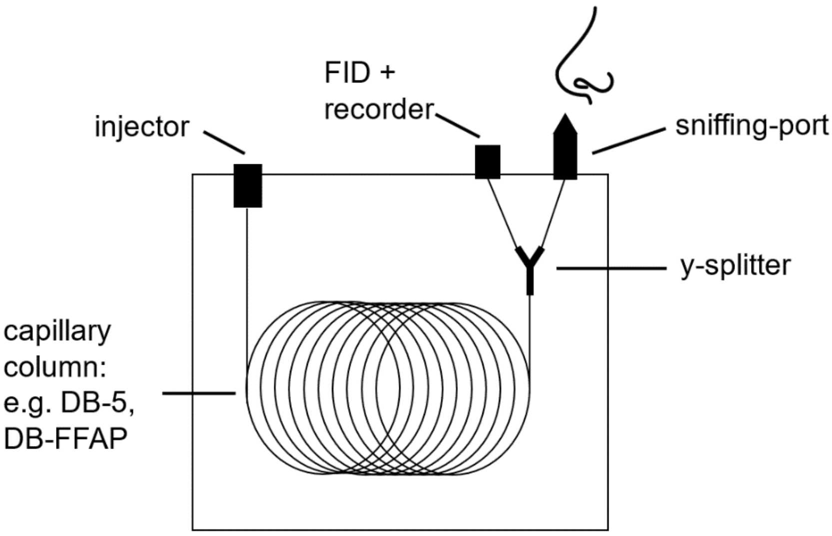


**Figure 6:** Schematic illustration of a GC-O system coupled with an FID and recorder.

Several methods have been developed for evaluating the relative influence of the individual odorants in the mixture of components. These include, among others, so called dilution-to-threshold procedures as described in aroma extract dilution analysis (AEDA) and combined hedonic aroma response measurement (CHARM) [132–134]. In this work, all GC-O analyses were performed in the way of an AEDA. Since this method has historically been applied to the analysis of foods, the method has been described using the term aroma [133]. However, this thesis deals with the analysis of vehicles which belong to consumer and non-food products of the everyday life. Consequently, the term of the method was modified to odor extract dilution analysis (OEDA).

During an OEDA, an undiluted odor extract or sample labeled with OD 1 (odor dilution) is diluted stepwise by a factor of 2 or 3 by volume (see Figure 7) [36, 133]. Each dilution is then analyzed by GC-O until no odor-active regions can be detected in the chromatogram. Thereby, the respective odor perceptions and descriptors are noted during the analyses. Finally, an overview is prepared showing the last dilution the odor was perceived (see Table 3). Those odorants which have the highest OD factors are assumed to have the highest impact on the overall odor of the sample [135].

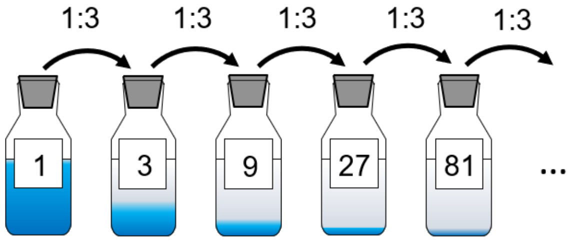


**Figure 7:** Dilution of a distillate according to OEDA.

**Table 3:** Example of an overview of the results of an OEDA.

| | OD 1 | OD 3 | OD 9 | OD 27 | OD 81 | OD factor |
|---|---|---|---|---|---|---|
| **grassy** | yes | yes | yes | no | no | **9** |
| **fatty** | yes | yes | no | no | no | **3** |
| **metallic** | yes | yes | yes | yes | no | **27** |
| … | | | | | | |

In the approach according to OEDA, the analysis is performed by at least two trained panelists on different capillary columns to avoid potential overlooking of odorants due to potential anosmia or coelution. However, the experience of the panelists is important for the analysis, as correct and unambiguous naming of the odorants is an essential requirement for subsequent identification of the odorants. There are also other methods for evaluating the relative influence of the individual odorants in the mixture of components such as direct intensity and detection frequency techniques, which will not be discussed further in this thesis [136, 137].

### 1.5.3 Identification and quantification of odorants in complex matrices

The data obtained by GC-O, such as odor quality, odor intensity, and OD factor allow only limited conclusions for an unequivocal identification. For a clear statement about the exact chemical structure of the odorants, a comparison of the analytical data with reference compounds is necessary [138]. Therefore, retention indices of the analytes with respect to the relative position of the homologous series of n-alkanes are calculated from the chromatographic data of the GC-O analysis [139, 140]. Since some compounds have similar chemical properties and can co-elute, retention indices on at least one further capillary column with different polarity are also determined. If databases already allow the conclusion on an odorant based on odor impression and retention indices, the substance can be tentatively identified by comparison of odor quality and intensity using GC-O. However, an unequivocal determination of the exact chemical structure is often only possible by means of mass spectrometric data, and alignment with the respective authentic reference compound [34].

Since the quantitatively dominating compounds in air samples of vehicle interiors are odorless, and many different volatiles are detectable, it is usually not possible to obtain mass spectra of odorants using standard analytical techniques such as GC-MS [5, 12]. This is also shown in analyses of complex polymer matrices, where odorless volatiles often co-elute with the targeted odorants [59]. The method of choice for solving co-elution is the application of two-dimensional gas chromatography-mass spectrometry (2D-GC-MS), as heart-cut or comprehensive 2D-GC-MS [141, 142].

In this work, odorants were identified using a heart-cut 2D-GC-MS/O system (see Figure 8; comprising an additional sniffing port). Thereby, a selected fraction of the effluent from the first GC system is transferred to a second GC system containing a capillary column with a different polarity [126]. The selected fraction is then separated again (see Figure 9; example of a chromatogram of vehicle interior emissions) and, if the concentration is high enough, a mass spectrum can be obtained, which can then be compared with entries in the NIST Mass Spectral Library. In the analysis of VOCs originating from non-food matrices, reference compounds are often not available for odorants. These must then be synthesized in a time-consuming and cost-intensive manner, or preparative isolated analyte fractions are subjected to further investigations such as $^{1}$H-NMR or $^{13}$C-NMR measurements, allowing conclusions regarding unique structural information [85].

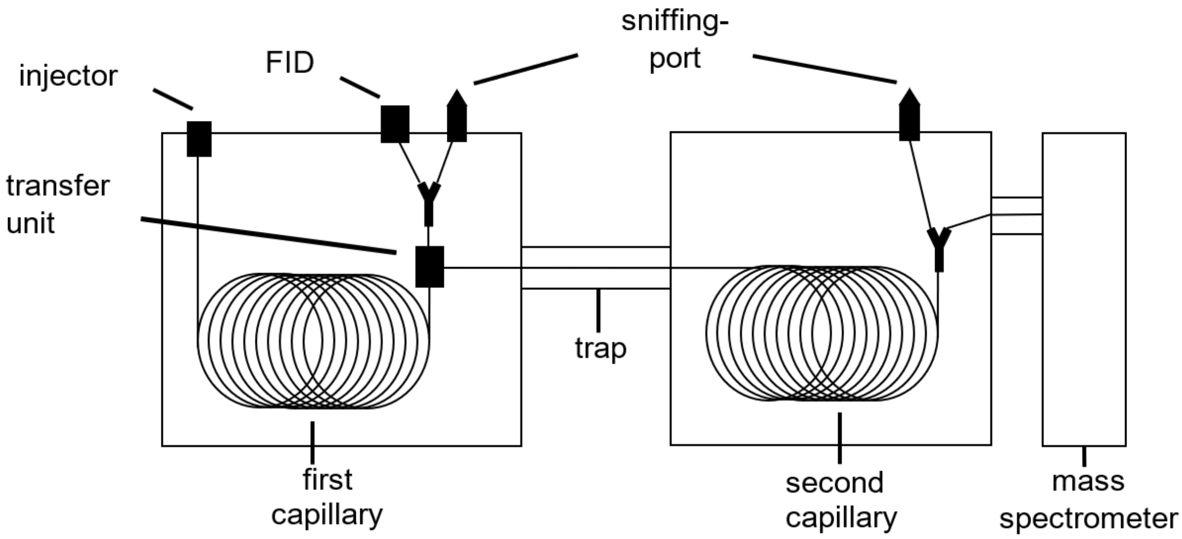


**Figure 8:** Schematic illustration of a heart-cut 2D-GC-MS/O system.

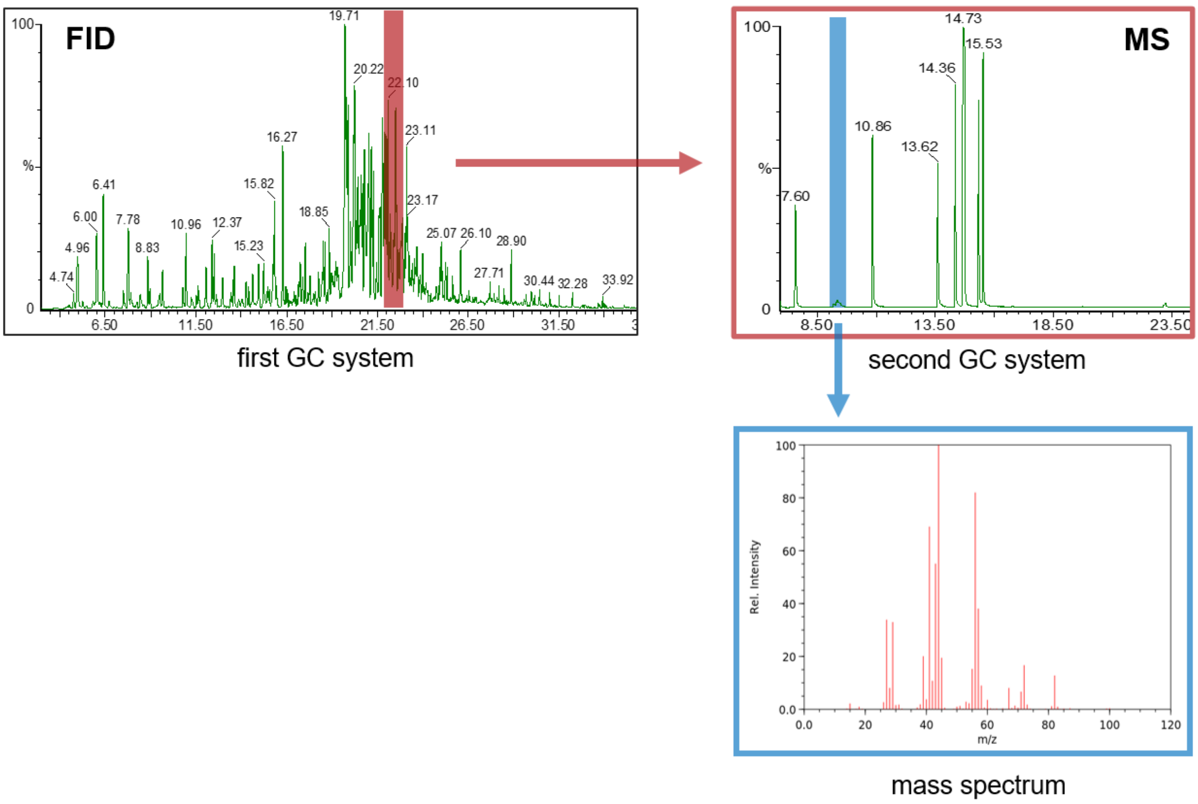


**Figure 9:** Separation of a selected fraction of the effluent of a vehicle interior emission sample on a second GC system by means of a heart-cut 2D-GC-MS/O system with the mass spectrum of hexanal recorded in EI mode. Data of mass spectrum: [143].

Quantitative investigations of odorants are often difficult to realize due to frequent co-elution and the challenge that most potent odorants are often just present in small amounts in most non-food materials. Especially measurements with an external calibration often do not provide reliable quantification results [144]. Therefore, stable isotope dilution assays for odorant quantification are found in the literature of aroma and smell research comprising non-food products such as plastics, being considered the most accurate method for quantification [145–147]. In this approach, stable isotopic labeled analogs of the respective odorants are added as an internal standard during sample preparation. Potential degradation reactions and losses of analytes can therefore be compensated as standards and analytes have almost identical physical and chemical properties. Since the use of isotopically labeled standards may be cost-intensive, non-labeled internal standards as isomeric compounds are often used, as applied in chapter 6 [148].

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# 2 Aims and outline

Vehicle interiors of newly produced cars are characterized by typical “new car” smells. These characteristic smell impressions are usually noticeable for several months to years and differ in intensity and, at times, character depending on the vehicle manufacturer. However, in contrast to the assumption that new car smell is specifically created in a laboratory, the odor is often a mere product of chance. Many different materials are equipped in the vehicle interior, which may release volatile compounds and odorants. So far, only limited information is available on the specific components responsible for the new car odor. Therefore, the overall objective of this thesis was to specifically characterize the odors in new cars and to reveal the respective potential sources of smell.

Time-consuming and cost-intensive investigations by the automotive industry are mainly related to the identification of potentially toxic or irritant compounds and the analysis of (odorless) main components of the gas phase. However, as quantitatively dominating VOCs do not automatically represent the main odor contributors, the nature of such causative odorants can only be elucidated by a combined sensory-analytical approach. Therefore, this work should focus on the analysis of volatile compounds by means of advanced gas chromatographic-mass spectrometric analyses coupled with human sensory evaluation, allowing a comprehensive analysis of odor-active compounds. Not only visible and large-surface materials of the vehicle interior can have an influence on the odor in the passenger cabin, however, volatile compounds from the vehicles’ body can also enter the passenger cabin through air bridges and could there influence the odor. Consequently, this work should focus on the investigation of the odor of a non-visible functional material in the vehicle body. Considering that some materials are directly produced at the vehicle production site and may change their physical or chemical properties during/ after vehicle assembly, this work should additionally investigate the odor change of a functional material due to hardening after vehicle production as part of a simulation experiment.

Previous investigations of odorants of non-food materials in literature were primarily carried out by extraction using solvents, thermodesorption of very small specimens or the enrichment/ concentration of volatile compounds in small gas volumes. A targeted investigation of odorants of complex vehicle components or large assemblies is only possible by breakdown and multiple analyses. Consequently, the aim of this work should be to develop sampling procedures for the characterization of odorants by means of emission test chamber measurements allowing a comprehensive analysis of the odorants in only one step. In in a further project, the developed sampling procedure should be applied to investigate whole passenger cabins. Due to the interfering and uncontrollable factors of the ambient air, standardized sampling of vehicle interiors is difficult. To overcome this challenge, a sampling procedure should be developed to investigate vehicle interiors in a controlled environment in a whole vehicle test stand for interior emissions. Accordingly, the aim of this work was to frame a proof-of-principle study, demonstrating that whole car analysis is possible, and reveals novel insights, given the respective technical and analytical procedures. In addition, there is a considerable knowledge gap how the composition of odorants in the vehicle cabin changes during vehicle use. Furthermore, the insights about the influence of temperature and ventilation on the decay/ change of the odor in the vehicle cabin is currently limited. Therefore, another aim of this work was to frame a case study demonstrating that investigation of the changes in gas phase of the vehicle interior is achievable and to gain knowledge for the improvement of the VIAQ and the sustainable reduction of unwanted odor nuisances in vehicles.

In chapter 3 and 4, the odor of an aqueous cavity preservation should be investigated as the smell of the rust prevention agent in the cavities of the vehicle body was perceived in the vehicle interior. The results should be discussed in relation to other studies focusing on the odorants of a passenger cabin and the odor change after hardening of the cavity preservation should be determined. In chapter 5, the odorants of the aqueous cavity preservation should be studied with additional methods by means of emission test chamber measurements. Thereby, two different sensory evaluation methods and three different sampling procedures for the characterization of odorants should be compared. In chapter 6, vehicle interiors of two new cars with different seat upholsteries should be investigated directly after vehicle delivery in a whole vehicle test stand for interior emissions via descriptive sensory analysis, identification, and quantification of potent odorants. Finally, in chapter 7, two vehicles should be subjected to everyday use by a customer. At specific time intervals during use, the odorant composition of the vehicle interiors should be determined by sampling in the whole vehicle test stand by olfactometer and descriptive sensory analysis and identification of the odor potency of the odorants. All studies are designed to gain knowledge on the odor composition in passenger cabins, with the overarching objective to control the vehicle interior regarding their emissions and odors, to meet the increasing consumer’s expectation of an odorless or neutral ("good") smelling car, and to raise the comfort in the mobility sector in general.

# 3 Volatile compounds in the vehicle-interior: Odorants of an aqueous cavity preservation and beyond

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# 4 Investigations on the impact of hardening on the odour of an aqueous cavity preservation for automotive applications using sensory and instrumental analysis

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# 5 Odor characterization of a cavity preservation using emission test chambers by different sensory evaluation methods and sampling concepts for instrumental analysis

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# 6 Follow your nose - traveling the world of odorants in new cars

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# 7 Smells like new car or rather like an old carriage? - Resolution of the decay behavior of odorants in vehicle cabins during usage

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# 8 Conclusion and outlook

In this thesis, more than 60 different odorants in the environment of new cars were successfully characterized. Thereby, a corrosion inhibitor for an automotive application was investigated regarding its odor for the first time. The aspect that odorants inside the vehicle body can enter the vehicle interior via air bridges has not been considered in scientific investigations yet. However, targeted combinatory sensory and instrumental analyses of odorants of the liquid and hardened cavity preservation revealed odorants which were most likely to stem from oxidation processes. However, this phenomenon of the formation of odorants via oxidation has already been observed in several non-food products and should therefore be considered in future material development.

Furthermore, the odorants of the cavity preservation could be successfully detected utilizing different sampling protocols involving emission test chambers. Thereby, the results showed that the sampling protocols can be applied to investigate the odorous constituents of a whole passenger cabin in only one step without the need of a complete breakdown of all materials and components of interest. The results build the foundation for future research on the odorant composition of the vehicle interior and allow the access to sampling from challenging vehicle parts or assemblies with a strong or unpleasant odor in emission test chambers. In this context, it is important to mention that also bulky products from living rooms or the everyday live can be investigated by means of the developed sampling concepts, which allow a future targeted development of improved manufacturing processes.

In a proof of principle study, odorants of two new car interiors with different specifications were investigated with a previously developed sampling protocol. Thereby, several odor-active compounds of different substance classes were identified and quantified. The aldehydes 2-butyl-2-heptenal, 2-propyl-2-octenal and (*Z*)-2-butyl-2-octenal showed high odor-potency and were reported for the first time as odorants in the environment of a passenger cabin. The formation mechanisms of the odorants allowed conclusions on the potential source of smell in the vehicle interior. Thereby, the findings implied that for reduction or even elimination of potent odorants, a large number of materials and components most likely need to be reconsidered and modified. Subsequently, the initially investigated vehicles were handed over to a customer and investigated in a case study by monitoring the causative odorants at defined time intervals after vehicle delivery and during use. However, the results demonstrated that investigation of the changes in gas phase of the vehicle interior is achievable and allowed to pin down the main influencing factors for development of a vehicle smell character that evolves over time. Additionally, the vehicle measurements showed a clear correlation between the decrease of the general emissions and odorants and elevated levels of temperature in the vehicle interior.

The results of this first study of the new car odor offer a big potential for future research projects. For example, different vehicle models from different manufacturers can be compared or cars with more differing equipment can be investigated. In addition, all odorants of the vehicle interior could be quantified to gain further insights into the composition of the gas phase. The findings could also be used to develop gas sensors or learning systems that facilitate odor quality control. Furthermore, there is a great need for research in analyzing the individual components or their source materials in more detail with regard to the release mechanisms of odorants. To date, this has been widely disregarded, but it will enable a better understanding of the origin and composition of odors in cars and the everyday live in general. All in all, a continuous improvement and sustainable development of material and products is necessary to raise the comfort in the mobility sector and to meet the requirements of higher consumer acceptance and enhanced wellbeing.